Bond Strength of Silorane- and Methacrylate-Based Composites to Resin-Modified Glass Ionomers

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DEDICATION

I dedicate this to my wife, Franzis. She has been my support throughout the program and ever since I met her. She's been there taking care of our daughters and myself when I've come home late night after night. I couldn't have completed this program without her. I love her and look forward to all the time we have together ahead of us. Sweetheart, thank you for making me the luckiest!

I also dedicate this to my daughters who love me still despite how often I was gone. I've loved seeing them grow up and am excited to be spending more time with them as they continue to grow and develop into beautiful women.

And I'd be remiss if I do not also honor my parents. I was incredibly blessed to have been raised by two loving and incredibly devoted parents. They raised me through their wonderful examples and gave me the gifts of confidence, a loving home, knowledge of the gospel and the opportunity and encouragement to learn.

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ABSTRACT

Objective: This study evaluated the shear-bond strength of a resin-modified glass-ionomer (RMGI) restorative material (Fuji II LC, GC Corporation) to a new silorane-based composite (Filtek LS, 3M/ESPE) and a methacrylate-based composite (Filtek Z250, 3M/ESPE) in a sandwich technique with various combinations of surface treatments and bonding agents. **Methods**: The RMGI was bonded to Filtek LS or Z250 using the proprietary adhesive (Filtek LS System Adhesive (LSA), 3M/ESPE) or methacrylate-based (Clearfil SE Bond (CSE), Kuraray) self-etch adhesive systems. Molds were filled with the RMGI and the surface was prepared in one of four ways: no treatment, etched with phosphoric acid, bur-roughened, or both bur-roughened and etched. Either the LSA or CSE adhesive system was applied according to the manufacturer's instructions. Next, either Filtek LS or Z250 was bonded to the RMGI, creating 16 groups of 10 specimens each. Specimens were stored for 24 hours at 37°C in 100% humidity and tested in shear (Instron). A mean and standard deviation was determined per group. A three-way ANOVA was used to evaluate the effect of surface preparation (4-levels) and bonding agent (2-levels) on the shear-bond strength of composite (2-levels) to RMGI (alpha=0.05). **Results:** A significant difference in bond strength was found based on composite ($p<0.001$) and bonding agent ($p<0.001$) but not on surface treatment ($p=0.699$) with no significant interactions ($p>0.05$) (see Table 1). **Conclusions:** Surface modifications did not affect the shear-bond strength of the silorane or methacrylate composites to the RMGI. The new silorane composite (Filtek LS) had significantly lower bond strength to the RMGI compared to the methacrylate composite (Filtek Z250). The

new silorane system adhesive agent (Filtek LS System Adhesive) had significantly higher bond strength to the RMGI compared to the methacrylate adhesive agent (Clearfil SE Bond). The greatest bond strengths to the RMGI were produced when using the silorane system adhesive agent with the methacrylate composite.

Table 1- Mean Shear Bond Strength

Resin Composite	Bonding Agent	Mean Shear Bond Strength MPa (st dev)			
		No modification	Etch only	Bur only	Bur and etch
Filtek LS	LSA	10.11 (5.68)	8.82 (5.43)	10.23 (4.41)	12.05 (2.46)
	CSE	4.13 (4.15)	6.89 (5.65)	7.23 (5.32)	4.09 (3.17)
Filtek Z250	LSA	18.13 (5.80)	15.56 (5.63)	14.71 (4.40)	15.68 (4.08)
	CSE	15.36 (2.26)	12.84 (2.59)	11.22 (2.93)	12.52 (4.15)

TABLE OF CONTENTS

Page

Title i

Approval.....ii

Dedication.....iii

Acknowledgements.....iv

Abstract.....v

Table of Contents.....viii

List of Figures x

List of Tables xi

I. BACKGROUND AND LITERATURE REVIEW

A. A Brief History of Glass Ionomers and Resin Composites 1

B. Polymerization Shrinkage and How it Affects Glass Ionomers and Resin Composites 2

C. Introduction of the Sandwich Technique and Resin-modified Glass Ionomers 3

D. Development of a Silorane-based Composite.....6

II. OBJECTIVES

A. Objective Overview.....9

B. Specific Hypotheses 11

III. MATERIALS AND METHODS

A. Experimental Design Overview 13

B. Experimental Design 13

C. Statistical Management of Data.....33

IV. RESULTS.....34

V. DISCUSSION

A. Significant differences between Filtek LS and Filtek Z250.....37

B. Significant differences between Filtek LS System Adhesive and Clearfil SE Bond 40

C. No significant differences between various surface modifications.....48

VI. CONCLUSION 50

VII. APPENDIX

A. Raw Data by Group.....51

B. Statistical Analaysis.....60

Literature Cited 65

LIST OF FIGURES

Page

Figure 1 RMGI Mold Fabrication.....17

Figure 2 Preparation of Fuji II LC for Groups #1, 2, 5, 6, 9, 10, 13, 14 18

Figure 3 Preparation for Fuji II LC for Groups #3, 4, 7, 8, 11, 12, 15, 16....19

Figure 4 Bonding of Either Z250 or Filtek LS to Fuji II LC 21

Figure 5 Shear Bond Strength Testing 27

Figure 6 SEM of Prepared Surfaces.....28

Figure 7 SEM of Fracture Mode 30

Figure 8 Mean Bond Strength 35

Figure 9 Fracture Mode.....36

Figure 10 Photos of Filtek LS System Adhesive and Filtek LS.....42

LIST OF TABLES

Page

Table 1 Mean Shear Bond Strength vii

Table 2 Study Materials.....14

Table 3 Study Groupings.....15

I. BACKGROUND AND LITERATURE REVIEW

A. A Brief History of Glass Ionomers and Resin Composites

In 1972, Wilson and Kent (1972) introduced to the dental market the first glass-ionomer cement. The original glass ionomers became well-known for their fluoride-releasing property (Forsten, 1977, Maldonado et al., 1978, Swartz et al.,1984), biocompatibility (Nakamura et al., 1983, Lan et al., 2003), and ability to chemically bond to hydroxyapatite (Causton and Johnson, 1979, Bowen, 1992, Erickson and Glasspoole 1994, Mount, 1994), but their overall strength, wear resistance, esthetics and multiple other physical properties were not adequate for use in many stress-bearing regions of the mouth (Erickson and Glasspoole, 1994, Mount, 1989).

The traditional methacrylate-based dental composites were first developed in the mid 1960s as a replacement for silicate cements and unfilled resins (Bowen, 1965). Since then, they have greatly improved in properties and handling characteristics such that now many providers consider it a primary restorative material (Eklund, 2010, Nascimento et al., 2010). At the outset, some of the physical properties were lacking, such as color stability (Powers et al., 1978), and wear resistance (Leinfelder, 1987). Over the years these properties have improved greatly with the changes made to the initiator, introduction of microfiller particles, and hybridization of manufacturing processes.

B. Polymerization Shrinkage and How it Affects Glass Ionomers and Resin Composites

A few properties continue to hinder the methacrylate-based composite resins; foremost among them is polymerization shrinkage. The average methacrylate-based composite resin restorative material shrinks approximately 3% (Feilzer et al., 1988, Erickson and Glasspoole, 1994, Kleverlaan and Feilzer, 2005). The direction of polymerization shrinkage has been shown to be toward the bonded surfaces, contrary to the previous belief that it was toward the light source (Versluis et al., 1998, Cho et al., 2002). Still, that compensation does not seem adequate to overcome the overall stress generated (Ferracane, 2008). Polymerization shrinkage has been cited to cause stress within the tooth as opposing walls are pulled toward each other. These stresses generated by the shrinkage can overcome the weaker bond to dentinal surfaces versus the stronger bond to enamel, thereby reducing the contraction stresses within the tooth by causing a gap at the dentin/restoration interface (Feilzer et al., 1987, Kinomoto and Torii, 1998, Kinomoto et al., 2000, Ferracane, 2008). This shrinkage and the resultant gap formation at the dentin interface has been thought to be a major cause for marginal microleakage and the resultant failure of composite restorations due to secondary caries, though that has yet to be definitively proven (Ferracane and Mitchem, 2003, Larson, 2005, Ferracane, 2008). Enamel/composite margins are typically free from this defect due to the much greater bond strength seen between composite and enamel than to dentin (Yazici et al., 2007). This could, in part, explain the results gathered by Mjör (1998), which showed the vast majority of recurrent caries occur at the gingival

margins, and suggests the need for a better bond at the dentinal/restoration interface.

Although glass ionomers have also been shown to have an approximate volumetric shrinkage of 3%, the important difference is the time it takes to polymerize. An example of a glass ionomer used by Feilzer et al. (1988) showed that it took over 5 minutes for the shrinkage to occur vs. the near instantaneous shrinkage seen with resin composite materials (Naoum et al., 2011). The rate at which a material is polymerized has been cited to be a greater factor in determining the amount of contraction stress, rather than the material (Kinomoto et al., 1999, Ferracane, 2008). Thus, since glass ionomers take longer to polymerize, the stresses generated are not as deleterious to the restoration/tooth interface.

C. Introduction of the Sandwich Technique and Resin-modified Glass Ionomers

In 1985, McLean et al. (1985) proposed a technique which attempted to combine the best of two worlds. It became known as the sandwich technique. The technique used by McLean et al. is now called the closed sandwich technique. It was revisited and advocated in 1990 by Suzuki and Jordan (1990). Since then a slight variant, called the open sandwich, has been introduced. Both techniques utilize the dentinal bond that glass ionomers can achieve by placing a layer of that material at the

gingival margin of the preparation. The difference between the two methods is based on the extent of the glass ionomer placement. In the closed technique, the glass ionomer does not extend completely to the cavosurface margin, but rather it leaves space for the composite resin to seal the margin. The open technique extends the glass ionomer to the cavosurface margin. For both, the remainder of the preparation is then restored with a composite resin to provide superior overall strength, wear resistance, enamel bond strength, color matching and other physical characteristics.

The closed sandwich technique has been shown to have a significantly larger gap formation and greater microleakage than the open technique (Reid et al., 1994, Aboushala et al., 1996). The open sandwich technique also showed significantly less microleakage than the composite resins, and proved that the sandwich technique could possibly be used to overcome the resin's shortcoming of polymerization shrinkage and the resultant dentin marginal gap formation (Aboushala et al., 1996, Friedl et al., 1997, Dietrich et al., 1999, Hagge et al., 2001, Besnault and Attal, 2003). However, not all studies agree. Ausiello et al. (1999) showed better marginal integrity using resin-based composites than with the sandwich technique on extracted endodontically treated molars. This study, though, did not indicate whether an open or closed sandwich technique was used and hints that the dentin may have been desiccated prior to RMGI bonding.

The initial drawback to the sandwich technique, however, was the interface between the conventional glass ionomer and composite resin. Glass ionomers do not form a chemical bond with the resins and must, therefore, be acid etched to create a micro-mechanical bond with the resin (McLean et al., 1985, Mount, 1989, Subrata and Davidson, 1989).

In the late 1980s, resin was added to the glass ionomers, and this hybrid genre was given the name of resin-modified glass ionomers (RMGI) (Antonucci et al., 1988). The addition of resin improved many of the drawbacks of glass ionomers. The resin also allows the material to be light activated, with a command set. The earlier set of the resin protects the glass ionomer portion from dehydration during the acid/base reaction seen with traditional glass ionomers (Feilzer et al., 1995, Sidhu and Watson, 1995). This curing sensitivity had previously plagued the glass ionomers making them difficult to handle. The addition of the resin also improved the strength characteristics (Mitra, 1991) while still retaining the glass ionomer's ability to release fluoride (Mitra, 1991, Momoi and McCabe, 1993, Sidhu and Watson, 1995) and bond chemically to dentin. Sidhu (1994) showed that RMGIs had less dentinal marginal gap formation than did a conventional glass-ionomer, and Sjödin et al. (1996) showed that RMGIs had less microleakage than conventional glass ionomers. The strength, wear resistance, and enamel bond did not approach that of a traditional resin composite, but was a definite improvement over the conventional glass ionomers (Uno et al., 1996). It also greatly improved the bond strength to other resin

composites, solving the initial drawback of the sandwich techniques. With the advent of RMGI, the open sandwich technique became less technique sensitive and had a better bond between the RMGI and resin composite (Kerby and Knobloch, 1992, Sidhu and Watson, 1995, Bona et al., 2007). This technique has been reviewed and used successfully for many years now (Andersson-Wenckert et al., 2004).

D. Development of a Silorane-based Composite

Recently, a unique composite, Filtek LS, has been developed by 3M/ESPE. Instead of the traditional methacrylate-derived monomer, Filtek LS, utilizes a silorane monomer ring. Many recent and ongoing studies of this new material have shown it to be similar in many ways to the methacrylate-based composites. It demonstrates "relatively higher flexural strength/modulus, fracture toughness" (Lien and Vandewalle, 2010), wear resistance (Trempler et al., 2009), and it may even be less irritating to pulpal tissue (Kostoryz et al., 2006, Li et al., 2008). It's arguable, though, whether or not the bond strength to tooth structure is as good as the methacrylate-based composites (Thalacker et al., 2004, El-Shamy et al., 2010, Almeida E Silva et al., 2010, Yaman et al., 2010, Giacobbi and Vandewalle, 2010). However, it shares similar marginal integrity (Thalacker et al., 2005, Thalacker et al., 2009, Hooshmand, 2009, Gao et al., 2011) hinting that an open sandwich technique may still be advisable.

The distinct advantage that Filtek LS has over all the other methacrylate-based composites, though, is its reduced polymerization shrinkage. The methacrylate-chain monomers lose spatial dimension as they covalently bond together through the light-activated free-radical reaction, resulting in the typical 3% polymerization shrinkage. Polymerization shrinkage has been blamed for marginal microleakage and internal stresses placed on the opposing bonded tooth surfaces (Ferracane and Mitchem, 2003, Ferracane, 2008). The ring shape of the silorane monomer counteracts this shrinkage by expanding the ring into a linear dimension during the polymerization reaction. The expansion of the ring before polymerization has been shown to decrease the polymerization shrinkage to an average of 1-1.5% (Weinmann et al., 2005, Lien and Vandewalle, 2010). The decrease in shrinkage could be advantageous by reducing stresses on the tooth, although there are some conflicting reports (Min et al., 2010 vs. Marchesi et al., 2010). Min et al. found that Filtek P90 (3M/ESPE), the name under which Filtek LS is marketed in countries outside the United States, generated less polymerization stress than Filtek Z250 or Z350 (3M/ESPE) concluding that "the low-shrinkage silorane-based composite demonstrated considerable reduction in shrinkage strain and stress." Marchesi et al. found that the methacrylate-based resins Venus Diamond (Heraeus-Venus) and Tetric EvoCeram (Ivoclar Vivadent) generated less stress than Filtek LS, though confirmed that Filtek LS generated less stress than Filtek Z250. They concluded "that simply reducing the shrinkage does not ensure reduced stress development in composites."

Methacrylate-based composites faced two challenges -- dentinal bond strength and marginal microleakage; this was the basis for developing the sandwich technique. However, even with its reduced polymerization shrinkage, Filtek LS has not shown a significant improvement in these areas. The use of a sandwich technique, therefore, may still be indicated when using Filtek LS and the gingival margin is in dentin. The RMGIs utilized in this technique, however, are formulated with a methacrylate-based monomer which may not bond adequately to Filtek LS, negating the use of the sandwich technique.

II. OBJECTIVES

A. Objective Overview

No studies to date show whether or not RMGIs can be bonded to Filtek LS, as advocated in the open-sandwich technique described previously, though the placement instructions for Filtek LS do state “self-adhesive materials such as glass ionomer cements… or resin-modified glass ionomer cements… may be used as cavity liner or reliner.” (Filtek LS Instruction for Use). The RMGIs utilize a methacrylate-based monomer whereas Filtek LS uses a silorane monomer ring. De Goes et al. (2010) showed that a RMGI luting cement, RelyX Luting Plus Cement (3M/ESPE), bonded adequately to Filtek LS using the proprietary adhesive. Syrek et al. (2009) showed that Filtek LS can bond to Filtek Z250 using the proprietary adhesive and Filtek Z250 can bond to Filtek LS using a methacrylate-based resin. These studies indicate that the resin component of RMGIs should be able to bond with Filtek LS regardless, perhaps, of the type of bonding agent used. This current study evaluated the bond strength between a RMGI restorative material (Fuji II LC) and Filtek Z250 or Filtek LS using either the proprietary adhesive, Filtek LS System Adhesive, or a methacrylate-based adhesive, Clearfil SE Bond.

The study also examined the effect of surface preparation on the bond strength. Sometimes it is necessary to remove excess RMGI when performing the sandwich technique clinically. An example situation would be with the RMGI Fuji II LC which comes in a capsule whose tip can be too wide for a narrow preparation and cannot

be always placed in a thin layer in the gingival box or simply from the tendency to overfill. This may force bulk placement in the box and the need to reduce the material to allow completion of the sandwich restoration, in effect removing the air-inhibited layer. Another surface preparation would be the acid etching performed before composite placement. How do these affect the bond strengths? Clearfil SE Bond and the Filtek LS System Adhesive, which were used in this study, are sixth-generation, self-etching primer bonding agents. Although these bonding agents are sixth-generation, self-etching agents, some studies have shown that selectively etching the enamel with 37% phosphoric acid prior to the bonding agent application leads to improved bond strengths of sixth-generation agents when bonding to enamel (Erickson et al., 2009) and less marginal defects and discoloration (Peumans et al., 2007, Ermis et al., 2010). In composite repair studies, though, typically acid etching a methacrylate-based composite resin surface has little effect on surface roughness (El-Askary et al., 2009, Maneenut et al., 2010) and reduced repair bond strengths compared to bur-roughened surfaces (Rodrigues et al., 2009, Vivas et al., 2009, Yesilyurt et al., 2009). Also, one study showed that a methacrylate adhesive can be used to bond a methacrylate composite to Filtek LS, though it did not study whether a methacrylate adhesive could be used to bond Filtek LS to a methacrylate composite (Syrek et al., 2009). Another study, though, indicated that only the Filtek LS System Adhesive should be used for bonding to dentin (Brandt et al., 2008).

The results of this study should give evidence to clinicians who use Filtek LS as to whether or not the “sandwich technique” can be used and which surface preparation would result in a stronger bond. It may also clarify whether or not the proprietary Filtek LS System Adhesive must be used with Filtek LS.

B. Specific Hypotheses

This study tested three specific null hypotheses as follows:

1) There is no significant difference in the bond strength to Fuji II LC based on type of composite (Filtek Z250 or Filtek LS).
2) There is no significant difference in the bond strength to Fuji II LC based on type of adhesive bonding agent (Filtek LS System Adhesive or Clearfil SE Bond).
3) There is no significant difference in the bond strength to Fuji II LC based on surface preparation (no treatment, bur-roughened, etched, or bur-roughened and etched).

This study tested four alternative hypotheses as follows:

1) Filtek Z250 has a stronger bond to Fuji II LC than does Filtek LS.
2) Filtek LS has a stronger bond to Fuji II LC when using the Filtek LS System Adhesive versus Clearfil SE Bond.
3) Filtek Z250 has a stronger bond to Fuji II LC when using Clearfil SE Bond versus Filtek LS System Adhesive

4) Roughening Fuji II LC with a bur and etching the surface creates a stronger bond between Filtek LS and Filtek Z250 versus no treatment, simply etching or simply bur roughening.

III. MATERIALS AND METHODS

A. Experimental Design Overview

The materials which were used in this experiment are Fuji II LC, Filtek Z250, Filtek LS, Filtek LS System Adhesive and Clearfil SE Bond (see Table 2).

A total of 16 groups were created (see Table 3). Ten specimens were prepared per group resulting in 160 total specimens. Filtek LS (groups #1-8) or Filtek Z250 (groups #9-16) were bonded to Fuji II LC using the Filtek LS System Adhesive or Clearfil SE Bond with: 1) no surface treatment, 2) phosphoric-acid etchant only, 3) carbide bur surface preparation only, or 4) carbide bur surface preparation and phosphoric acid etchant. Shear bond strength was tested after 24 hours of storage in 100% humidity.

B. Experimental Design

All samples were created by one provider to minimize interoperator differences and to ensure uniformity of fabrication. A mold for the RMGI was formed by gluing a 7.9mm internal diameter and 1.5mm deep metal washer flush with one end of a 12.7mm diameter x 12.7mm high PVC pipe. The purpose of the PVC was to secure placement of the specimen within the Instron 5543 testing machine (Instron, Norwood, MA). A plastic disc, 11.1mm diameter x 0.75mm thick, was then glued to the internal side of the washer to prevent stone from entering the mold. The

Table 2- Study Materials

Material	Type	Manufacturer	Resin	Filler
Filtek LS	Silorane-based composite resin	3M/ESPE St. Paul, MN USA	Silorane	Quartz, yttrium fluoride
Filtek Z250	Hybrid methacrylate-based composite resin	3M/ESPE St. Paul, MN USA	Bis-GMA, Bis-EMA, UDMA, TEGDMA	Zirconia, silica
LS System Adhesive	Proprietary self-etching primer methacrylate-based bonding agent	3M/ESPE St. Paul, MN USA	HEMA; Bis-GMA	Silane-treated Silica
Clearfil SE Bond	Self-etching primer, methacrylate-based bonding agent	Kuraray, Kurashiki, Japan	HEMA; Bis-GMA	Silanated colloidal silica
Fuji II LC	Methacrylate-based resin-modified glass-ionomer	GC Corporation, Tokyo, Japan	HEMA; 2,2,4 TMHEDC; TEGDMA	Alumino-silicate glass
Gel Etchant	37.5% Phosphoric Acid gel	Kerr Orange, CA USA		

Table 3- Study Groupings

Substrate Material	Restorative Material	Bonding Agent	Surface Preparation	Group #
Fuji II LC	Filtek LS	LS System Adhesive	No modification	1
			Acid etch only	2
			Bur modification only	3
			Bur and acid etch	4
		Clearfil SE Bond	No modification	5
			Acid etch only	6
			Bur modification only	7
			Bur and acid etch	8
	Filtek Z250	LS System Adhesive	No modification	9
			Acid etch only	10
			Bur modification only	11
			Bur and acid etch	12
		Clearfil SE Bond	No modification	13
			Acid etch only	14
			Bur modification only	15
			Bur and acid etch	16

remainder of the PVC pipe was then filled with dental stone for structural strength (see Figure 1).

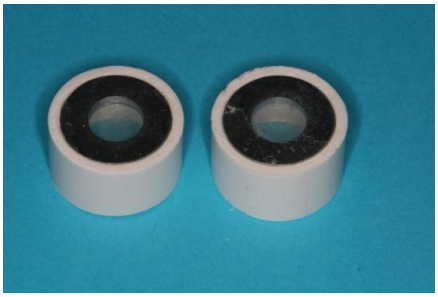
Fuji II LC was activated and mixed for 10 seconds in a triturator according to manufacturer's instructions. Once mixed, the RMGI was dispensed into the prepared mold. Immediately the surface of the Fuji II LC was flattened flush with the metal washer surface by sweeping a glass slide over the surface prior to curing, creating a smooth surface for groups 1, 2, 5, 6, 9, 10, 13, 14 (see Figure 2). This creates a smooth surface similar to intraoperative procedures to remove excess material. The RMGI was not cured through the glass slide, for this would have prevented the formation of an air-inhibited layer and this layer is desired since it is what would be created intraorally during a routine procedure. Once placed into the PVC/metal washer/stone mold, the Fuji II LC was light-cured for 20 seconds using a Bluephase 16i (Ivoclar) curing light. The curing light was monitored after every ten samples with a radiometer (LED radiometer, Kerr) to ensure it maintained a minimum light intensity of 1200mW/cm2 or greater.

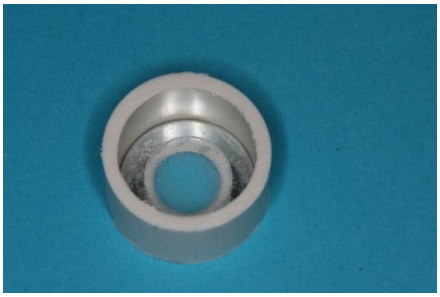
For the remaining groups 3, 4, 7, 8, 11, 12, 15, 16, the RMGI was slightly overfilled and then polymerized in the same manner as previously mentioned (see Figure 3). Overfilling the samples allowed for bur preparation back to a height of 1.5mm (i.e. flush with the metal washer surface) after polymerization, which also might be done

Figure 1- RMGI Mold Fabrication

A- The plastic disc glued to the internal surface of the washer

B- The metal washer glued into the PVC pipe

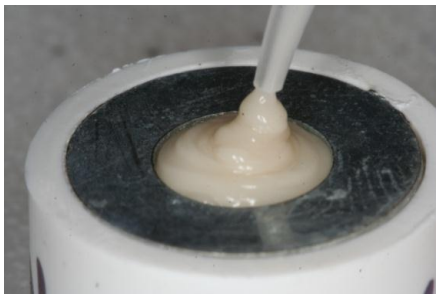




C- The remainder of the PVC pipe filled with Die Keen stone

Figure 2- Preparation of Fuji II LC for groups: 1, 2, 5, 6, 9, 10, 13, 14

A- Dispensing Fuji II LC into the mold



B- Flattening the surface with a glass slide

C- Light curing for 10 seconds

D- View of the final prepared surface

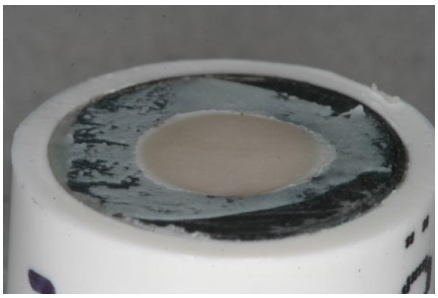
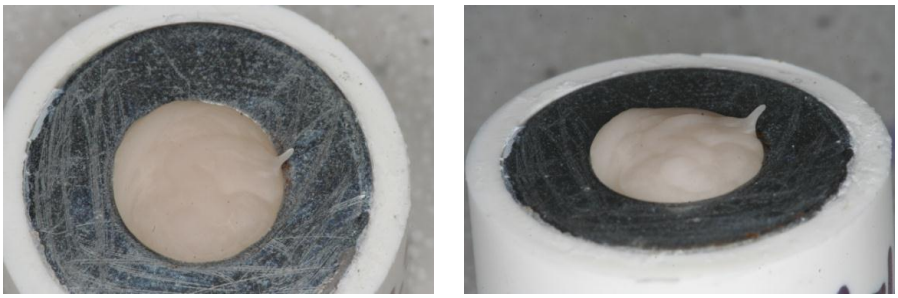


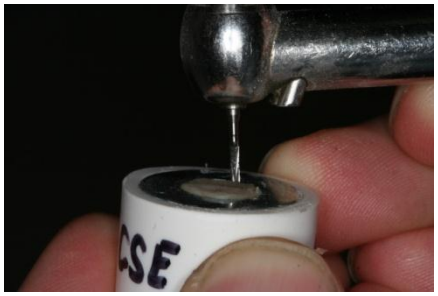
Figure 3- Preparation for Fuji II LC for Groups #3, 4, 7, 8, 11, 12, 15, 16

A- Overfilled mold with Fuji II LC

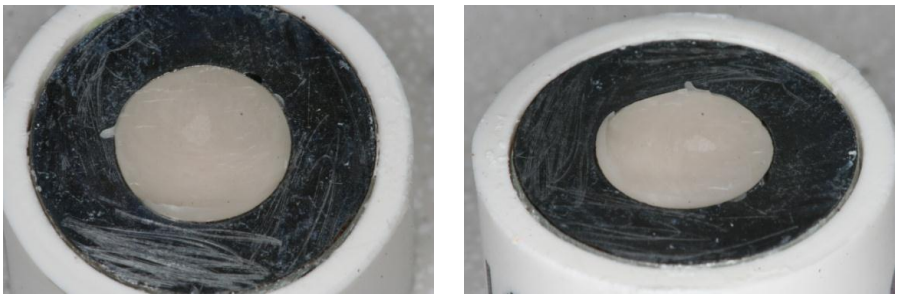


B- Light curing for 10 seconds

C- Preparation with a 557 carbide bur with water spray



D- View of the final prepared surface



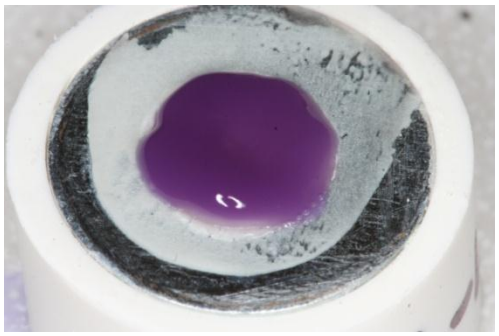
intraoperatively to remove excess material. A flat surface was also required in this study to remove a potential variable when comparing these samples to those flattened with a glass slide. The flat tip of a 557 carbide bur was used for this.

Prior to creating the study samples, the flattest surface was observed to be obtained on pilot samples by passing over the entire surface in one direction while holding the bur perpendicular to the RMGI surface. The sample was then turned ninety degrees and a second pass was performed over the entire surface, still holding the bur perpendicular to the surface (see Figure 3). Water spray was utilized during bur preparation to prevent desiccation of the RMGI. The bur was replaced after every ten specimens to ensure cutting efficiency.

The groups 2, 4, 6, 8, 10, 12, 14, 16 which were designated for phosphoric-acid etching were etched using Kerr Gel Etchant, 37.5% phosphoric acid (see Figure 4). The acid was placed for 10 seconds, consistent with recommendations for etching dentin prior to 3-step etch-and-rinse adhesive application. The instructions for Clearfil SE Bond advocate a 10-second etch of uncut enamel if desired and Filtek LS System Adhesive advocates 15 seconds (Clearfil SE Bond Instructions for Use, Filtek LS Instruction for Use). After 10 seconds of application time, the etch was rinsed and dried with 3 “brief bursts of dry, oil-free air” following the instructions of

Figure 4- Bonding of Either Z250 or Filtek LS to Fuji II LC

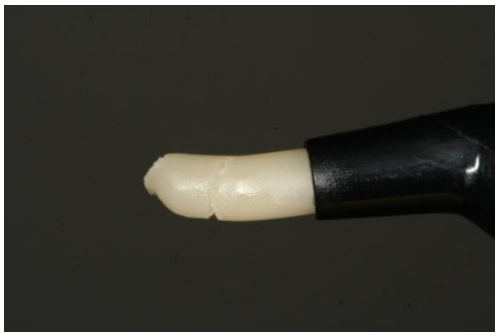
A- Etching of groups #2, 4, 6, 8, 10, 12, 14, 16



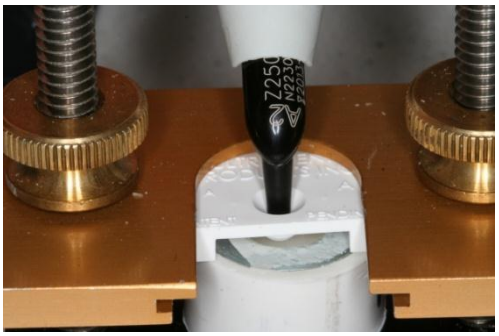
B- Bonding agent application

C- Placement of mold into the Ultradent jig

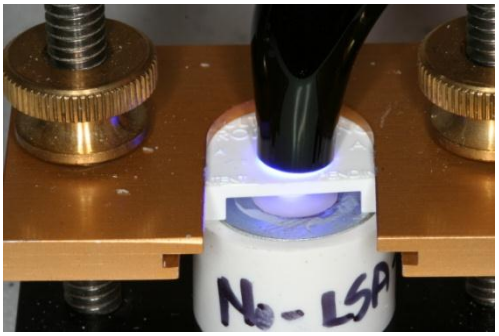
D- Expressing the initial 3-4mm of composite from Filtek LS due to the tendency for it to be dry and brittle

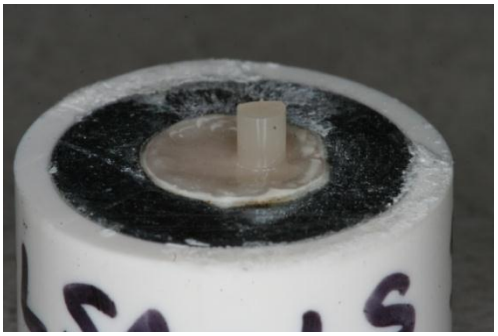


E- Placement of composite using the jig



F- Curing and Final Specimen





G- Storage case

Filtek LS System Adhesive (Filtek LS Instruction for Use). Clearfil SE Bond states to simply dry without further details.

Groups 1-4 and 9-12 had Filtek LS System Adhesive applied according to manufacturer's instructions. The steps used were the following. First, the bottle was agitated since the solution can become viscous if left stationary. Second, the self-etching primer was applied over the entire surface for 15 seconds then gentle air was used to create an even film over the sample. This layer was cured for 10 seconds using the Bluephase 16i curing light mentioned previously. Next, the adhesive was applied for 5-7 seconds covering the entire surface. The manufacturer's instructions do not specify a specific amount of application time for this step. Again, gentle air was used to create a uniform layer and the adhesive layer was cured for another 10 seconds (Filtek LS Instruction for Use). The respective composite for that test group was then immediately applied as described below (see Table 3).

In the remaining groups 5-8 and 13-16, Clearfil SE Bond was applied according to the manufacturer's instructions. The steps used were the following. First, the self-etching primer was applied over the entire surface and left for 20 seconds. Following the 20 seconds, a gentle stream of air was used to evaporate the solvent. Next, the adhesive was applied to the entire surface for 5-7 seconds. Again, the

manufacturer's instructions do not state an application time for this step. Gentle air was used to create a uniform layer and then cured for 10 seconds using the Bluephase 16i curing light. The respective composite for that test group was then immediately applied as described below (see Table 3).

After application and curing of the respective adhesive, the sample was placed into an Ultradent jig (see Figure 4). The jig contained a white, non-stick, 2.4mm diameter Delrin insert (Ultradent Corp.). The insert was stabilized against the RMGI and the composite placed 2.5mm in depth. It must be noted that the first 3-4mm of the Filtek LS had to be expressed from each compule prior to application of the insert. The first few millimeters were noted to be dry and brittle (see Figure 4). The composite was light-cured for 20 seconds using a Bluephase 16i (Ivoclar) curing light in agreement with the curing instructions for both Filtek LS and Filtek Z250 (Filtek LS Instruction for Use, Filtek Z250 Universal Restorative Instructions for Use).

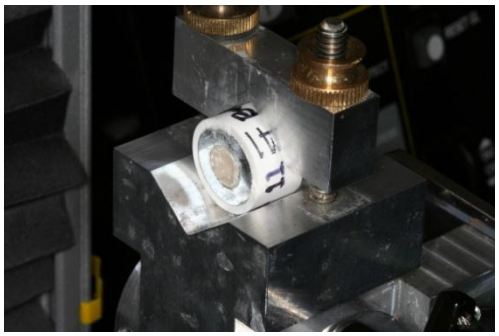
Upon removal from the jig, the specimens were placed in a sealed container with moistened paper towels to create 100% humidity and stored at 37°C in a lab incubator (Model 20 GC, Quincy Lab Inc., Chicago, IL). After 24 hours, the shear bond strength of all specimens was tested using the Instron 5543 testing machine (Instron, Norwood, MA) at a crosshead speed of 1mm/min using the notched blade

at a ninety-degree angle (see Figure 5). The resultant data of the various groups was then analyzed to verify the three null hypotheses.

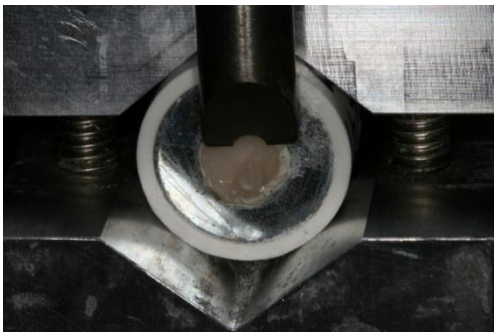
Following testing, the specimens were examined under a 10x microscope to determine the failure mode as either: 1) adhesive fracture at the adhesive interface, 2) cohesive fracture in the RMGI, 3) cohesive fracture in the composite, 4) mixed fracture (combination of adhesive and cohesive) in the RMGI, 5) mixed fracture (combination of adhesive and cohesive) in the composite, or 6) mixed fracture through the RMGI, adhesive, and composite. SEM images were made on examples of the different prepared surfaces and fracture modes (see Figures 6 and 7).

Figure 5- Shear Bond Strength Testing

A- Specimen within the Ultradent custom jig



B- Notched blade approximating the specimen prior to initiating test



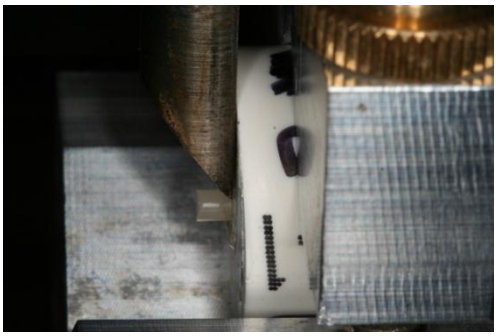
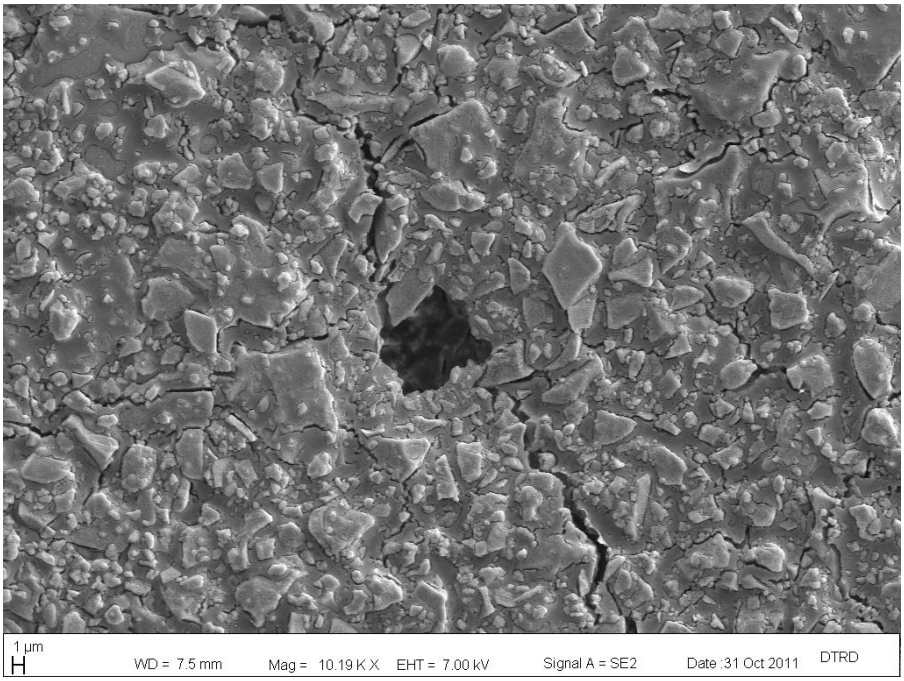
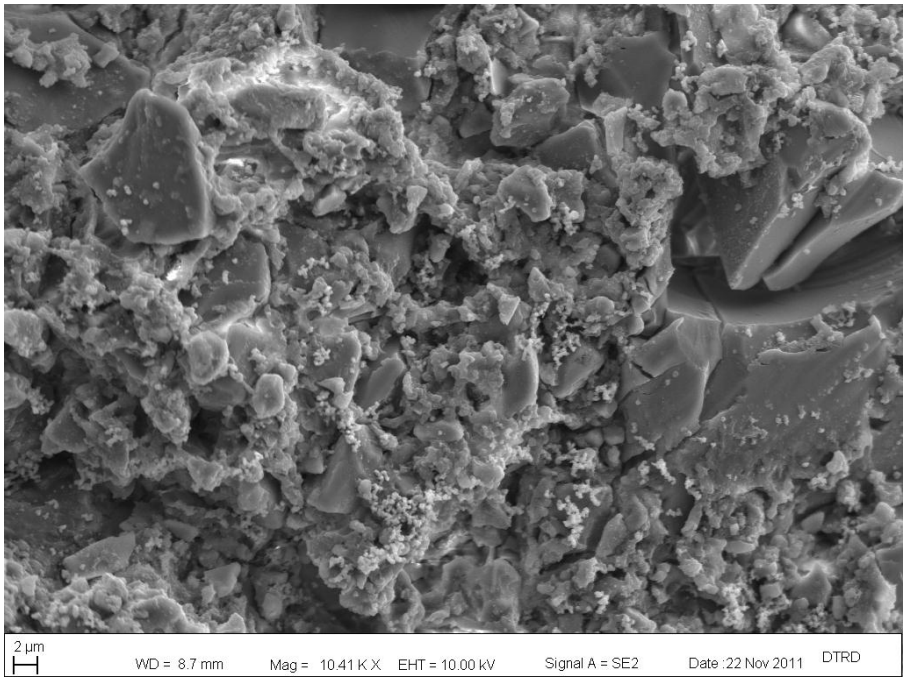


Figure 6- SEM of Prepared Surfaces (craze lines induced during preparation of specimen for SEM imaging)

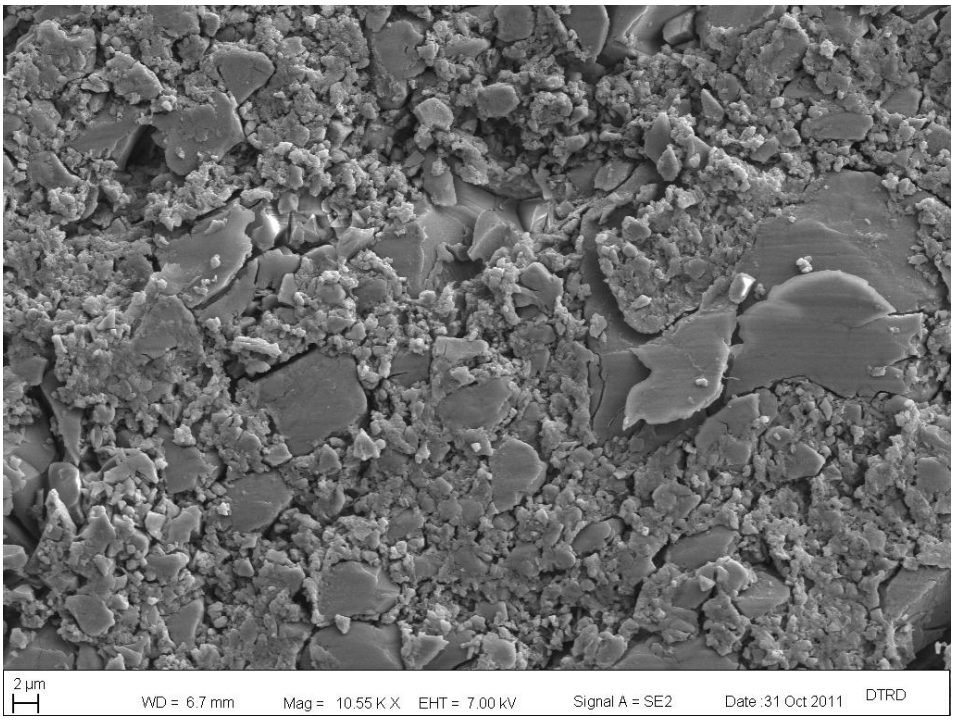
A- No surface treatment



B- Etched surface only



C- Bur-roughened surface only



D- Both bur-roughened and etched surface

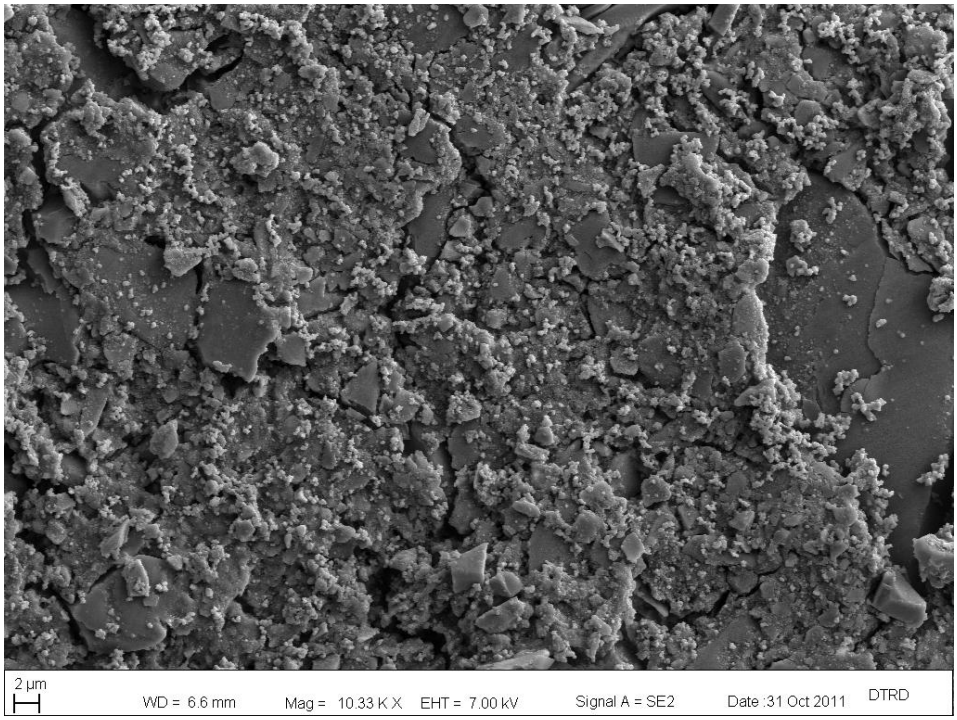
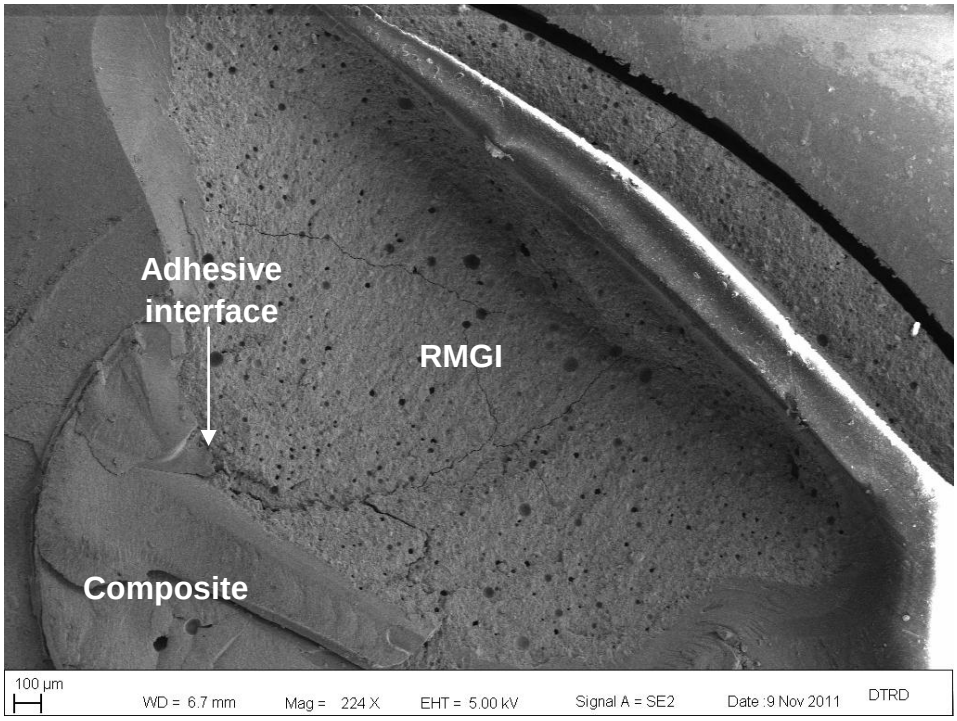


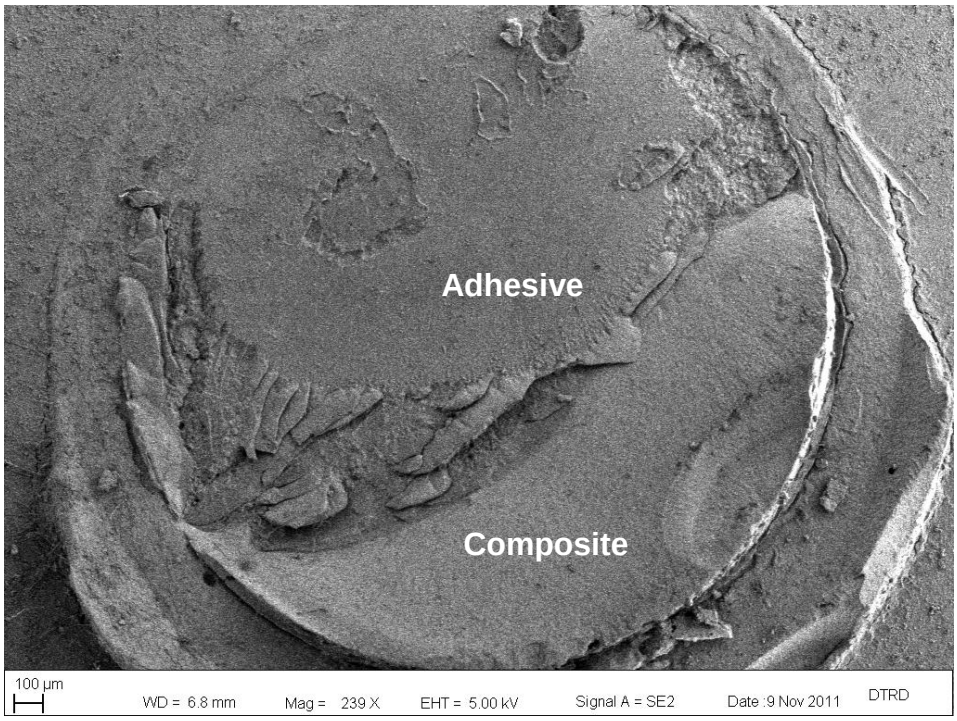
Figure 7- SEM of Fracture Mode (craze lines induced during preparation of specimen for SEM imaging)

A- Mixed fracture mode through all three substrates: RMGI, adhesive and composite

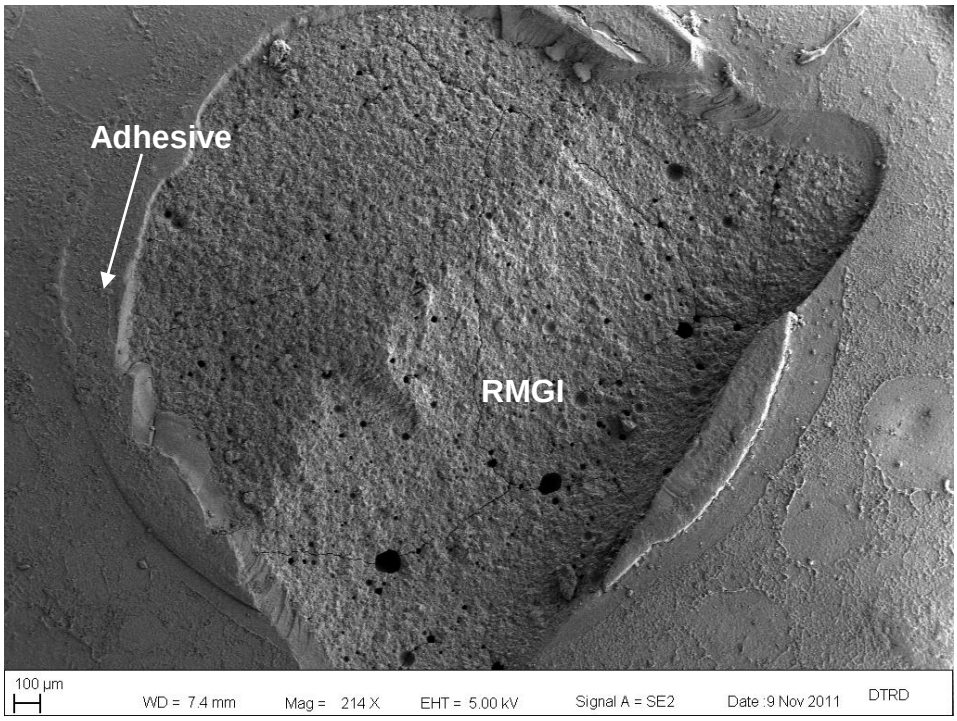


B- Cohesive fracture mode within the composite. (No images are available for this fracture mode since it was not evident within the samples of this study.)

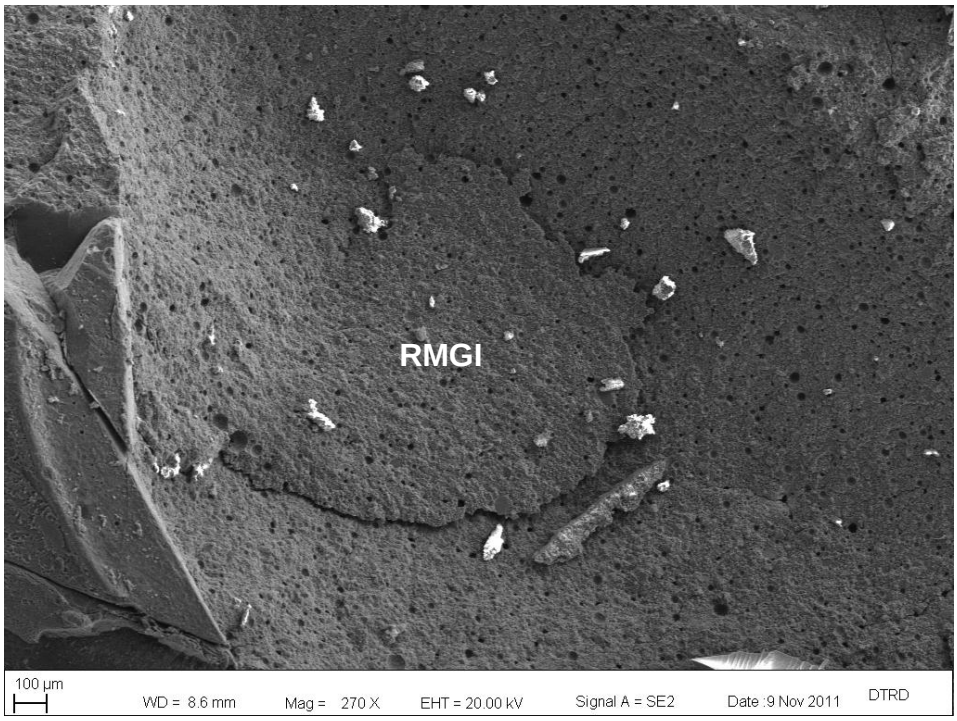
C- Mixed fracture mode in both the adhesive and composite



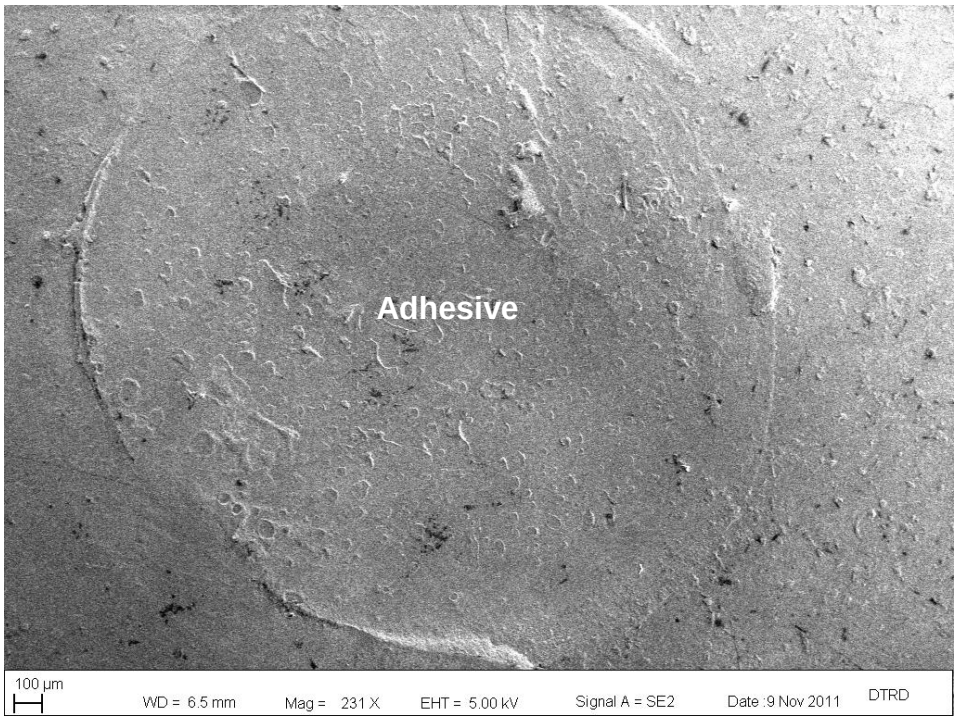
D- Mixed fracture mode in both the adhesive and RMGI



E- Cohesive fracture mode within the RMGI



F- Adhesive fracture mode only



C. Statistical Management of Data

A mean and standard deviation was determined per group. A three-way ANOVA was used to evaluate the effect of surface preparation with four levels (no treatment, etched, bur-roughened, and both bur-roughened and etched) and bonding agent with 2 levels (Clearfil SE Bond and Filtek LS System Adhesive) on the shear-bond strength of composite with 2 levels (Filtek Z250 and Filtek LS) to RMGI (alpha=0.05). See Appendix B.

An a priori power analysis showed that the sample size of 10 per group provided 80% power to detect small-effect size differences (0.223 to 0.264, or approximately 0.45 to 0.52 standard deviation difference) among means of the main effects when testing with a three-way ANOVA with 4, 2, and 2 levels at an alpha level of 0.05. The effect size for the interaction terms is similar (NCSS PASS 2002).

No significant interactions were seen after reviewing the ANOVA, thus the need for further statistical analysis was not required.

IV. RESULTS

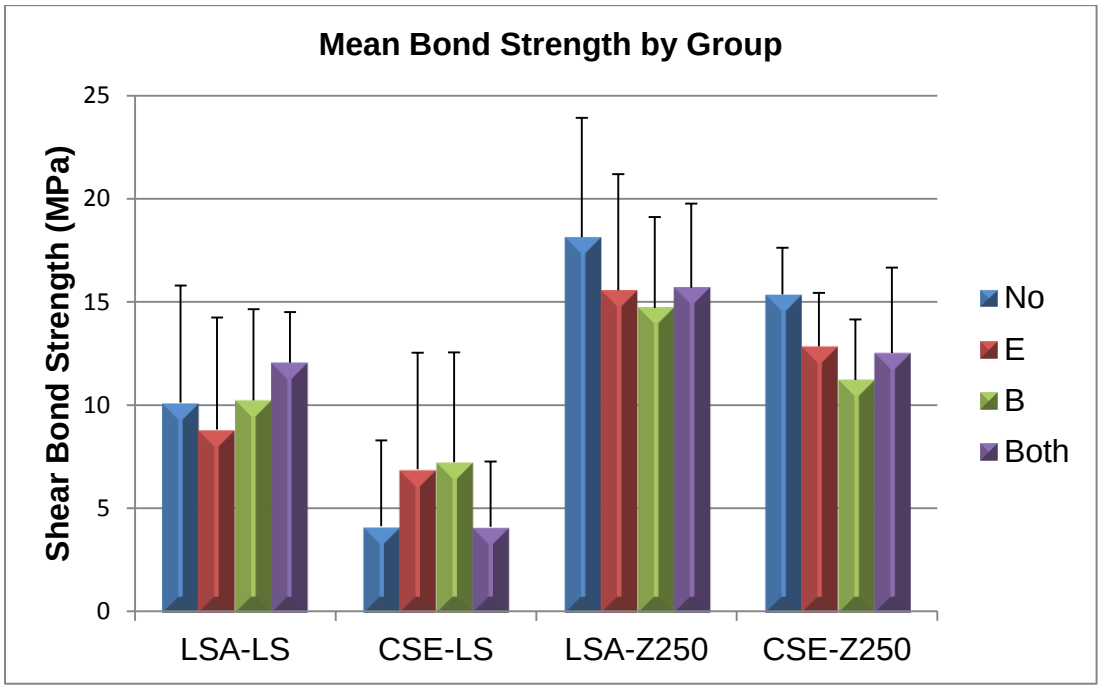
The statistical analysis was reviewed and approved by the clinical research administrator, Clinical Research Division, Lackland AFB, TX. A significant difference in bond strength was found based on composite ($p<0.001$) and bonding agent ($p<0.001$) but not on surface treatment ($p=0.699$) with no significant interactions ($p>0.05$) (see Figure 8 and Appendix B).

Since there were only two possible composites and two possible bonding agents, a Tukey's post-hoc test was not performed. By looking at the megapascal results, Filtek Z250 (14.50 MPa) had a significantly stonger overall shear bond strength than did Filtek LS (7.94 MPa) (see Appendix B).

This effect is also evident in the fracture mode. A failure within the adhesive layer was seen the majority of the time (68%) with Filtek LS irregardless of adhesive or surface treatment. For Filtek Z250, however, the failures were almost exclusively (90%) cohesive within the RMGI restorative material (see Figure 9).

Likewise, Filtek LS System Adhesive (13.16 MPa) had a significantly stronger shear bond strength than did Clearfil SE Bond (9.28 MPa) (see Appendix B). The difference is seen more clearly when trying to bond with Filtek LS.

Figure 8- Graph of Mean Bond Strength

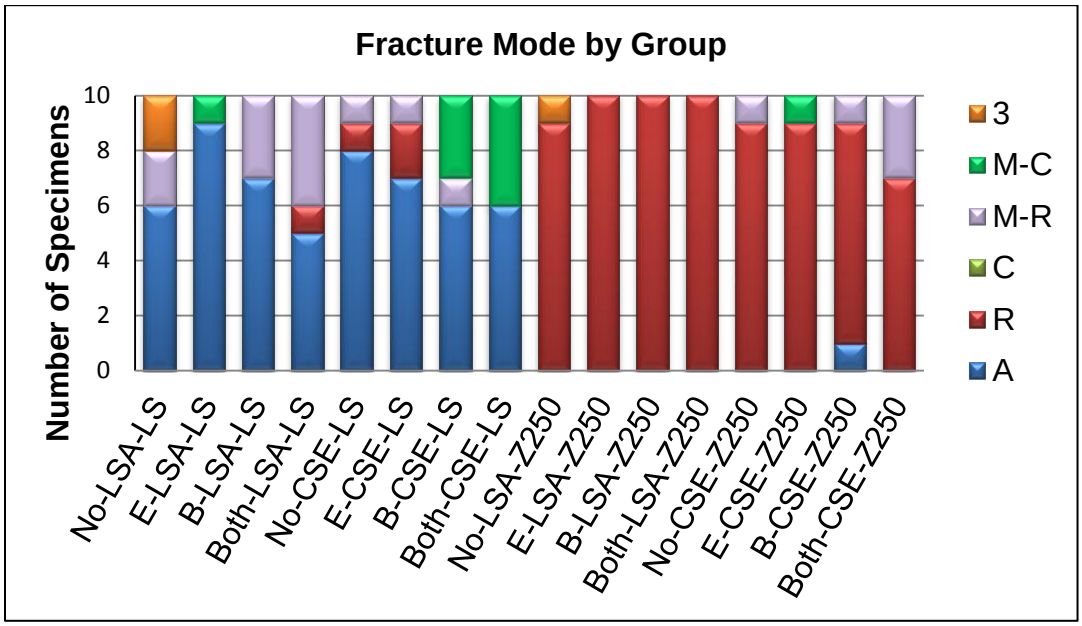


Legend

Treatment groups:
LSA: Filtek LS System Adhesive
LS: Filtek LS
CSE: Clearfil SE Bond
Z250: Filtek Z250

Surface treatment:
No: No surface treatment of RMGI
E: Etched only treatment of RMGI
B: Bur-roughened only treatment of RMGI
Both: Bur-roughened and etched treatment of RMGI

Figure 9- Graph of Fracture Mode



Legend

Treatment groups:
LSA: Filtek LS System Adhesive
LS: Filtek LS
CSE: Clearfil SE Bond
Z250: Filtek Z250

Surface treatment:
No: No surface treatment of RMGI
E: Etched only treatment of RMGI
B: Bur-roughened only treatment of RMGI
Both: Bur-roughened and etched treatment of RMGI

Fracture mode:
A: Adhesive fracture mode only
R: Cohesive fracture mode within the RMGI
C: Cohesive fracture mode within the composite
M-R: Mixed fracture mode in both the adhesive and RMGI
M-C: Mixed fracture mode in both the adhesive and composite
3: Mixed fracture mode through all three substrates: RMGI, adhesive and composite

V. DISCUSSION

Although the manufacturer's instructions for Filtek LS state that "self-adhesive materials such as glass ionomer cements… or resin-modified glass ionomer cements… may be used as cavity liner or reliner," (Filtek LS Instruction for Use) the results of this study would advise against such use, irrespective of bonding agent. The remainder of this section will address the null hypotheses and discuss the results.

A. Significant differences between Filtek LS and Filtek Z250

The first null hypothesis was rejected in this study. It stated that there would be no significant difference in the bond strength to Fuji II LC based on type of composite (Filtek Z250 or Filtek LS). This study, however, showed a significant difference in the bond strengths between these two materials.

As mentioned earlier during the literature review section, it is arguable whether or not the bond strength to tooth structure of Filtek LS is as good as the methacrylate-based composites. No formal published articles could be found that studied this relationship. The following recent studies, though, were reported in International Association of Dental Research abstracts. Thalacker et al. (2004) showed that Filtek LS with H-Bond could create the same if not better shear bond strength to bovine enamel or dentin than some of the traditional methacrylate resins, including Filtek

Z250 with Adper Prompt L-Pop. An online search for H-bond adhesive yielded no results, though it is likely an abbreviation for Hermes Bond, a one-step precursor of Filtek LS System Adhesive (Mine et al., 2010). Yaman et al. (2010) examined premolars marked for extraction due to orthodontics, bonded either Filtek Supreme XT with Adper Single Bond or Filtek LS with Silorane System Adhesive, extracted the teeth a month later, sectioned the teeth and then submitted them to microtensile bond strength testing. Their results yielded no significant difference between the two composites. And again in a 2010 by Giacobbi and Vandewalle (2010), there was no significant difference noted in the initial microtensile bond strengths of Filtek LS with LS Adhesive and Clearfil Majesty Posterior with Clearfil SE Bond to dentin. However, El-Shamy et al. (2010) showed that Filtek Z250 with Single Bond had significantly better shear bond strength than Filtek LS with LS Adhesive to enamel and dentin. This was also reported in a microtensile bond strength study by Almeida E Silva et al. (2010). Their study compared Filtek Z250 and Filtek LS and showed that Filtek Z250 had significantly better dentinal microtensile bond strength. The abstract, though, did not specify which bonding agents were used. It is assumed that Filtek LS System Adhesive was used with the Filtek LS composite.

The polymerization shrinkage discrepancy between Filtek LS and Filtek Z250, which has been fairly well shown (Weinmann et al., 2005, Lien and Vandewalle, 2010) and discussed previously, was negated in this experimental set-up since there was only

one bonded surface and the direction of shrinkage would be toward the bonded surface. Thus any effect this may have had on the bond strength was not an issue.

There are a few studies, though, that have shown bonding of Filtek LS to a RMGI. One previously mentioned study was De Goes et al. (2010) who showed that a RMGI luting cement, RelyX Luting Plus Cement (3M/ESPE), bonded to an already cured sample of Filtek LS using the proprietary adhesive, though it was significantly weaker than the bond using LS system adhesive and RelyX ARC or the self-adhesive cement RelyX Unicem. However, the fact that the Filtek LS was the cured substrate to which the RelyX Luting Plus Cement was applied may yield a different result than trying to bond Filtek LS to pre-cured RelyX Luting Plus Cement due to the curing chemistry. A more recent study by Boushell et al. (2011) showed that Filtek LS/LS System Adhesive had significantly equivalent shear bond strengths to Vitrebond Plus (3M/ESPE) as did Filtek Z250/Adper Scotchbond SE (3M/ESPE). The RMGI in this study was Fuji II LC, a restorative material rather than a luting cement or liner. Perhaps the difference seen in this study is related to the type of RMGI used. More studies could be performed to look at this specifically.

B. Significant differences between Filtek LS System Adhesive and Clearfil SE Bond

The second null hypothesis was also rejected. It stated that there would be no significant difference in the bond strength to Fuji II LC based on type of bonding agent (Filtek LS System Adhesive or Clearfil SE Bond). This study showed a significant difference in bond strengths due to the dental adhesive used. The failure mode also showed the majority of failures in the Filtek LS group were adhesive in nature, whereas the Filtek Z250 groups had a high majority of failures being cohesive in nature within the weaker RMGI material. Not only were the failures adhesive, but the Filtek LS samples were associated with lower bond strength, larger standard deviations and thus a larger coefficient of variability suggesting a weaker interface. The cohesive failures of the Filtek Z250 samples indicate the opposite, a more stable interface (Frankenberger et al., 2001). This can also be seen in four samples from the Filtek LS group which immediately failed upon testing with one other sample testing at near zero MPa. This failure at the interface, especially seen in the Filtek LS groups bonded with Clearfil SE Bond, reflects part of the results seen in a study by Brandt et al. (2008) which showed that no bond formed between Filtek LS and other tested methacrylate-based bonding agents, with the exception of Filtek LS System Adhesive. Since the majority of the failures between Filtek LS and the RMGI, Fuji II LC, were adhesive in nature, it is prudent to look at the respective dental adhesives and how they compare.

Filtek LS System Adhesive is advocated by 3M ESPE to be the only bonding agent to be used with Filtek LS. In the Filtek LS technical product profile, it makes multiple references to the exclusive use of Filtek LS with its specified adhesive. An example of such references are, “The formulation of LS System Adhesive specifically fits the chemistry of the Filtek LS restorative (Filtek LS technical product profile, 2007).” Their rationale is that:

> Due to its siloxane backbone, the silorane resin is more hydrophobic than conventional methacrylate resins, so it results in reduced water uptake and related phenomena, as described in the Test Results section. That means this adhesive has to bridge a larger difference between the hydrophilic tooth substrate and the hydrophobic silorane material as compared to conventional methacrylate materials (Filtek LS technical product profile, 2007).

This exclusive use of these two products together is even printed on their individual containers (see Figure 10). But how truly different is Filtek LS System Adhesive to Clearfil SE Bond?

The Clearfil SE Bond was chosen specifically for this study based on the fact that it, like Filtek LS System Adhesive, is a bonding agent which utilizes a self-etching primer, also known as two-step self-etch. It has also been shown in multiple studies to have consistently stronger bond strengths than other two-step self-etch bonding agents and equivalent to those of the three-step etch-and-rinse (Velasquez et al., 2006, Sarr et al., 2010, Heintze et al., 2010) and superior clinical longevity (Peumans et al., 2005). The differences between Filtek LS System Adhesive and Clearfil SE Bond seem to be

Figure 10- Photos of Filtek LS System Adhesive and Filtek LS- illustrating the exclusive use of the adhesive and composite resin restorative material as system

slight, thus Clearfil SE Bond was chosen for a comparison, but they will be looked at in depth.

At a macro level there are very few differences noted except in their application method. As described in the experimental design set-up section, Filtek LS System Adhesive prescribes massaging the self-etch primer "over the entire area for 15 sec… use a gentle stream of air… [and] cure the Primer for 10 seconds…" The bonding agent is then applied, a gentle stream of air used and again cured for ten seconds. This application method differs from that of Clearfil SE Bond which applies the primer for twenty seconds rather than fifteen, and does not cure the primer before placement of the bond. One study has shown that increasing the application time (10 seconds to 30 seconds) can significantly increase shear bond strengths when averaging multiple types of adhesives including Clearfil SE Bond, but does not address if this holds true specifically for Clearfil SE Bond alone (Velasquez et al., 2006). No similar study, however, was found for Filtek LS System Adhesive for comparison. There were also no studies found when an online search was performed that compared the bond of Filtek LS System Adhesive or Clearfil SE Bond with and without curing between steps. Because of the lack of studies, it cannot be known whether or not these differences of primer application time or primer curing may have an effect in Filtek LS System Adhesive having a significantly better bond strength than did Clearfil SE Bond in this study.

This study demonstrated, though, that both bonding agents could be used to bond Filtek Z250 to the RMGI. In fact, the Filtek LS System Adhesive outperformed Clearfil SE Bond. From an abstract by Brandt et al. (2008), they also demonstrated that the Filtek LS System Adhesive could bond multiple methacrylate resins. The study by De Goes et al. (2010) also showed the ability of Filtek LS System Adhesive to bond to other methacrylate-based restorative materials. These results would refute the statement by 3M/ESPE that Filtek LS System Adhesive should only be used with Filtek LS. In fact, in this study it showed a significantly stronger bond than did Clearfil SE Bond. Perhaps modifiying the bonding agent to create a more hydrophobic layer also improves the bond to methacrylate-based composites.

Neither bonding agent, however, could adequately bond Filtek LS to the RMGI, though Clearfil SE Bond did produce a significantly lower bond with Filtek LS than did Filtek LS System Adhesive. Still, this portion of the results would indicate that the largest discrepancy is not between the RMGI and the bonding agent, but the bonding agent and Filtek LS. This could perhaps also be the explanation seen in the results of the El-Shamy et al. (2010) and Almeida E Silva et al. (2010) studies mentioned previously. To pursue this possibility, a closer look at the chemistry behind the two adhesives is warranted.

In general, bonding agents work by creating a free radical that can attack a susceptible bond within a monomer. This creates a small polymer with a free radical end and the reaction continues until there is no available substrate for reaction due to depletion or physical space limitations. The overall effect is a polymer chain reaction that is the source of a composite's strength. Both the Clearfil SE Bond and the Filtek LS System Adhesive utilize this basic reaction.

When reviewing the MSDS for both materials, both contain methacrylate-based resin monomers and neither contain silorane monomers. The basic chemistry appears fairly similar. They both try to span the gap between the necessary hydrophilic layer closest to the dentin and a hydrophobic resin composite. The differences seem to be pH, a few of the monomers (though still methacrylate-based), and filler.

Filtek LS System Adhesive has a higher pH (2.7 versus 2.0), and states that the acidic monomer helps "initiate the ring-opening cationic cure of Filtek LS restorative, thus providing chemical bonding to Filtek LS." (Filtek LS Technical Product Profile) If it is simply the acidic nature of the monomer, then Clearfil SE Bond, which is more acidic, should be able to also initiate the ring-opening action for bonding to Filtek LS. The acidic nature of the bonding agent, therefore, doesn't seem to be the differentiating factor, unless Clearfil SE Bond is too acidic and thus cannot aid in the initiation of Filtek LS.

Filtek LS System Adhesive primer uses a phosphoric acid-methacryloxy-hexylesters, Bisphenol A diglycicyl Ether Dimethacrylate (Bis-GMA), 2-Hydroxyethyl Methacrylate (HEMA), and the Vitrebond copolymer seen in many of their RMGI formulations. The adhesive uses HEMA, Triethylene glycol dimethacrylate (TEGMA) and the phosphoric acid-methacryloxy-hexylesters. The Clearfil SE Bond primer also uses an acidulated phosphate monomer, called MDP (10-Methacryloyloxydecl dihydrogen phosphate), as well as HEMA, and a dimethacrylate monomer. The adhesive uses the same components as the primer with the addition of BisGMA. Both materials use an acidulated phosphate monomer and contain only methacrylate-based monomers. The respective Vitrebond copolymer and MDP are also stated to serve the same purpose, which is to create a chemical bond to the dentinal collagen fibers, but this study did not bond to a dentin surface, rather a RMGI. Perhaps it is the vitrebond copolymer found within Filtek LS System Adhesive and its likeness to those within the RMGI that can explain the superior bond to Fuji II LC versus Clearfil SE Bond.

But how does this formulation give any advantage when bonding to Filtek LS? Both adhesives are methacrylate-based and utilize the same radical reaction for polymerization. Both would have similar methacrylate double bonds to react with the silorane monomer of Filtek LS. Both claim to create a hydrophobic superior layer requisite to bonding a resin composite. A study by Mine et al. (2010) states, “further details on how this methacrylate-based SSA-Bond (referring to Filtek LS System

Adhesive) links to the silorane composite is currently not known…" beyond the claim of a hydrophobic layer chemically compatible with Filtek LS mentioned previously. The difference in monomers still cannot adequately explain the inferior bond seen between Clearfill SE Bond and Filtek LS.

Lastly, Filtek LS System Adhesive states it uses a silane-treated silica filler particle with an average dimension of 7nm which is "finely dispersed to prevent settling (Filtek LS Technical Product Profile)." Clearfil SE Bond states that a silanated colloidal silica is used, though the dimension is not mentioned (Clearfil SE Bond Instruction for Use). In order to strengthen the cohesive strength of a composite it is necessary to chemically bond the filler to the resin matrix. This is accomplished frequently by silanating the filler (Craig and Powers, 2002). The silanated filler can improve the bond to both the RMGI and to the composite, but both adhesives use silanated fillers. Perhaps there is a difference in the chemical structure of the respective silane molecules used, or the size, shape or other dimension of the filler that could account for the significant difference noticed in their respective bond strengths.

Although this study cannot elucidate what the major difference is between Filtek LS System Adhesive and Clearfil SE Bond that allows one to adhere more effectively to Filtek LS than the other, it does confirm that Filtek LS System Adhesive generates a

significantly stronger bond with Filtek LS. Furthermore, it demonstrated that neither bonding agent could create a significantly equivalent bond strength of Filtek LS to RMGI versus Filtek Z250 to the RMGI.

C. No significant differences between various surface modifications

This study failed to reject the third null hypothesis. It stated that there would be no significant difference in the bond strength to Fuji II LC based on surface preparation (no treatment, bur-roughened, etched, or bur-roughened and etched). This study found no significant interaction between surface preparation and shear bond strength for either composite or dental adhesive.

The results of surface treatment modifications are consistent with previous studies. Bona et al. (2007) demonstrated that etching did not lessen microleakage between a RMGI and a composite. Maneenut et al. (2010) conducted a study in which two different RMGI cements were repaired and the surfaces were treated either with or without etching. No significant differences were noted in bond strength. Also, the SEM they performed “showed little effect from acid treatment.” Kerby et al. (1992) actually showed that acid etching worsened the bond between Fuji II LC and a composite resin. Roughening the surface with a bur was thought to potentially create a better bond due to increased surface area, but again the results of this study did not substantiate that theory. It does demonstrate, though, that removing

the air inhibited layer or accidentally etching the RMGI surface, as may be done clinically during the sandwich technique, does not worsen the bond.

VI. CONCLUSION

Surface modifications did not affect the shear-bond strength of the silorane or methacrylate composites to the RMGI. The new silorane composite (Filtek LS) had significantly lower bond strength to the RMGI compared to the methacrylate composite (Filtek Z250). The new silorane adhesive agent (Filtek LS System Adhesive) had significantly higher bond strength to the RMGI compared to the methacrylate adhesive agent (Clearfil SE Bond). The greatest bond strengths to the RMGI were produced when using the silorane adhesive agent with the methacrylate composite.

The recommendations, therefore, that can be given based on this study's results would be: 1) that surface modification of the RMGI is not needed to increase bond strength nor does it worsen it, 2) to use Filtek LS System Adhesive with Filtek LS, 3) to not utilize the sandwich technique with Filtek LS based upon its poor bond to a RMGI, Fuji II LC, and 4) that Filtek LS System Adhesive can bond to methcrylate-based composites.

Further studies could confirm these results by performing microleakage tests at the interface of RMGI and Filtek LS versus the interface of RMGI and Filtek Z250.

Appendix A- Raw Data by Group

Legend

Treatment groups:

LSA: Filtek LS System Adhesive

LS: Filtek LS

CSE: Clearfil SE Bond

Z250: Filtek Z250

Surface treatment:

No: No surface treatment of RMGI

E: Etched only treatment of RMGI

B: Bur-roughened only treatment of RMGI

Both: Bur-roughened and etched treatment of RMGI

Fracture mode:

A: Adhesive fracture mode only

R: Cohesive fracture mode within the RMGI

C: Cohesive fracture mode within the composite

M-R: Mixed fracture mode in both the adhesive and RMGI

M-C: Mixed fracture mode in both the adhesive and composite

3: Mixed fracture mode through all three substrates: RMGI, adhesive and composite

A- Group 1 (No-LSA-LS

Specimen	Newtons	Megapascals	Fracture Mode
1	12.21	2.77	A
2	0.04	0.01	A
3	28.95	6.56	M-R
4	72.18	16.37	3
5	43.85	9.94	A
6	42.92	9.73	A
7	73.39	16.64	3
8	58.38	13.24	A
9	68.97	15.64	A
10	44.99	10.20	M-R
	Mean	10.11	
	St Dev	5.68	
	%CV	56.23	

B- Group 2 (E-LSA-LS)

Specimen	Newtons	Megapascals	Fracture Mode
1	41.58	9.43	A
2	46.16	10.47	A
3	90.43	20.51	M-C
4	56.08	12.72	A
5	17.63	4.00	A
6	35.90	8.14	A
7	24.72	5.61	A
8	14.09	3.20	A
9	11.87	2.69	A
10	50.45	11.44	A
	Mean	8.82	
	St Dev	5.43	
	%CV	61.52	

C- Group 3 (B-LSA-LS)

Specimen	Newtons	Megapascals	Fracture Mode
1	64.53	14.63	M-R
2	35.72	8.10	A
3	61.49	13.94	A
4	53.82	12.20	A
5	7.24	1.64	M-R
6	39.47	8.95	A
7	22.44	5.09	A
8	41.77	9.47	M-R
9	64.28	14.58	A
10	60.31	13.68	A
	Mean	10.23	
	St Dev	4.41	
	%CV	43.08	

D- Group 4 (Both-LSA-LS)

Specimen	Newtons	Megapascals	Fracture Mode
1	48.72	11.05	A
2	63.98	14.51	M-R
3	37.37	8.47	A
4	56.38	12.78	M-R
5	52.62	11.93	A
6	47.10	10.68	A
7	71.84	16.29	R
8	48.19	10.93	M-R
9	63.91	14.49	A
10	41.35	9.38	A
	Mean	12.05	
	St Dev	2.46	
	%CV	20.44	

E- Group 5 (No-CSE-LS)

Specimen	Newtons	Megapascals	Fracture Mode
1	33.49	7.59	A
2	25.43	5.77	A
3	4.94	1.12	R
4	17.88	4.06	A
5	0.00	0.00	A
6	0.00	0.00	A
7	20.95	4.75	A
8	57.38	13.01	M-R
9	21.94	4.97	A
10	0.00	0.00	A
	Mean	4.13	
	St Dev	4.15	
	%CV	100.51	

F- Group 6 (E-CSE-LS)

Specimen	Newtons	Megapascals	Fracture Mode
1	9.21	2.09	A
2	12.82	2.91	A
3	55.54	12.59	R
4	23.85	5.41	A
5	62.91	14.26	R
6	21.16	4.80	A
7	11.88	2.69	A
8	77.04	17.47	M-R
9	12.72	2.88	A
10	16.82	3.81	A
	Mean	6.89	
	St Dev	5.65	
	%CV	82.00	

G- Group 7 (B-CSE-LS)

Specimen	Newtons	Megapascals	Fracture Mode
1	18.13	4.11	A
2	15.45	3.50	A
3	63.82	14.47	M-C
4	23.15	5.25	M-C
5	64.80	14.69	M-R
6	0.00	0.00	A
7	38.07	8.63	A
8	11.66	2.64	A
9	23.66	5.37	A
10	60.01	13.61	M-C
	Mean	7.23	
	St Dev	5.32	
	%CV	73.66	

H- Group 8 (Both-CSE-LS)

Specimen	Newtons	Megapascals	Fracture Mode
1	12.42	2.82	A
2	47.66	10.81	A
3	8.64	1.96	A
4	25.82	5.86	M-C
5	5.88	1.33	A
6	19.16	4.35	A
7	12.50	2.83	M-C
8	7.95	1.80	M-C
9	5.72	1.30	M-C
10	34.54	7.83	A
	Mean	4.09	
	St Dev	3.17	
	%CV	77.64	

I- Group 9 (No-LSA-Z250)

Specimen	Newtons	Megapascals	Fracture Mode
1	57.98	13.15	R
2	63.14	14.32	R
3	39.19	8.89	R
4	69.31	15.72	R
5	103.51	23.47	R
6	78.97	17.91	R
7	110.86	25.14	R
8	116.25	26.36	R
9	64.05	14.52	R
10	96.33	21.84	3
	Mean	18.13	
	St Dev	5.80	
	%CV	31.99	

J- Group 10 (E-LSA-Z250)

Specimen	Newtons	Megapascals	Fracture Mode
1	63.30	14.35	R
2	91.67	20.79	R
3	40.66	9.22	R
4	78.41	17.78	R
5	89.87	20.38	R
6	95.60	21.68	R
7	56.09	12.72	R
8	77.90	17.66	R
9	17.21	3.90	R
10	75.34	17.08	R
	Mean	15.56	
	St Dev	5.63	
	%CV	36.19	

K- Group 11 (B-LSA-Z250)

Specimen	Newtons	Megapascals	Fracture Mode
1	65.59	14.87	R
2	98.91	22.43	R
3	50.03	11.34	R
4	60.87	13.80	R
5	47.20	10.70	R
6	72.61	16.46	R
7	39.07	8.86	R
8	66.25	15.02	R
9	54.36	12.33	R
10	93.95	21.30	R
	Mean	14.71	
	St Dev	4.40	
	%CV	29.92	

L- Group 12 (Both-LSA-Z250)

Specimen	Newtons	Megapascals	Fracture Mode
1	47.98	10.88	R
2	87.04	19.74	R
3	65.61	14.88	R
4	96.77	21.94	R
5	49.12	11.14	R
6	72.93	16.54	R
7	77.88	17.66	R
8	85.31	19.34	R
9	64.06	14.53	R
10	44.81	10.16	R
	Mean	15.68	
	St Dev	4.08	
	%CV	26.02	

M- Group 13 (No-CSE-Z250)

Specimen	Newtons	Megapascals	Fracture Mode
1	59.89	13.58	M-R
2	65.97	14.96	R
3	62.52	14.18	R
4	62.14	14.09	R
5	54.72	12.41	R
6	84.62	19.19	R
7	63.41	14.38	R
8	70.12	15.90	R
9	84.62	19.19	R
10	69.43	15.74	R
	Mean	15.36	
	St Dev	2.26	
	%CV	14.68	

N- Group 14 (E-CSE-Z250)

Specimen	Newtons	Megapascals	Fracture Mode
1	62.93	14.27	R
2	65.14	14.77	R
3	53.50	12.13	R
4	46.37	10.51	R
5	47.58	10.79	R
6	56.93	12.91	R
7	44.27	10.04	R
8	76.59	17.37	M-C
9	69.00	15.65	R
10	43.79	9.93	R
	Mean	12.84	
	St Dev	2.59	
	%CV	20.21	

O- Group 15 (B-CSE-Z250)

Specimen	Newtons	Megapascals	Fracture Mode
1	55.98	12.69	R
2	48.29	10.95	M-R
3	41.39	9.39	R
4	24.59	5.58	A
5	71.74	16.27	R
6	38.40	8.71	R
7	47.34	10.74	R
8	53.95	12.23	R
9	52.75	11.96	R
10	60.50	13.72	R
	Mean	11.22	
	St Dev	2.93	
	%CV	26.11	

P- Group 16 (Both-CSE-Z250)

Specimen	Newtons	Megapascals	Fracture Mode
1	44.54	10.10	M-R
2	40.72	9.23	M-R
3	93.03	21.09	R
4	72.12	16.35	R
5	43.11	9.77	R
6	43.29	9.82	R
7	65.54	14.86	R
8	33.29	7.55	M-R
9	64.59	14.65	R
10	52.12	11.82	R
	Mean	12.52	
	St Dev	4.15	
	%CV	33.12	

Appendix B- Statistical Analysis

3-way ANOVA

Notes		
Output Created		09-AUG-2011 08:37:12
Comments		
Input	**Data**	E:\Nuttall data\3-way anova.sav
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	Weight	<none>
	Split File	<none>
	N of Rows in Working Data File	160
Missing Value Handling	**Definition of Missing**	User-defined missing values are treated as missing.
	Cases Used	Statistics are based on all cases with valid data for all variables in the model.
Syntax		UNIANOVA mpa BY agent comp surface /METHOD = SSTYPE(3) /INTERCEPT = INCLUDE /POSTHOC = surface (TUKEY) /PRINT = DESCRIPTIVE /CRITERIA = ALPHA(.05) /DESIGN = agent comp surface agent*comp agent*surface comp*surface agent *comp*surface .
Resources	**Elapsed Time**	0:00:01.07

Between-Subjects Factors		
		N
AGENT	**CSE**	80
	LSA	80
COMP	**LS**	80

	Z	80
SURFACE	**both**	40
	bur	40
	etch	40
	no	40

Descriptive Statistics
Dependent Variable: MPA

AGENT	COMP	SURFACE	Mean	Std. Deviation	N
CSE	LS	both	4.0880	3.1741	10
		bur	7.2280	5.3243	10
		etch	6.8921	5.6513	10
		no	4.1275	4.1486	10
		Total	5.5839	4.7390	40
	Z	both	12.5248	4.1488	10
		bur	11.2230	2.9307	10
		etch	12.8367	2.5948	10
		no	15.3614	2.2550	10
		Total	12.9865	3.3144	40
	Total	both	8.3064	5.6264	20
		bur	9.2255	4.6580	20
		etch	9.8644	5.2552	20
		no	9.7445	6.6160	20
		Total	9.2852	5.5121	80
LSA	LS	both	12.0509	2.4636	10
		bur	10.2283	4.4068	10
		etch	8.8190	5.4256	10
		no	10.1106	5.6847	10
		Total	10.3022	4.6359	40
	Z	both	15.6804	4.0807	10
		bur	14.7127	4.4014	10
		etch	15.5564	5.6303	10

		no	18.1311	5.8009	10
		Total	16.0202	5.0060	40
	Total	**both**	13.8657	3.7722	20
		bur	12.4705	4.8649	20
		etch	12.1877	6.3957	20
		no	14.1209	6.9409	20
		Total	13.1612	5.5909	80
Total	**LS**	**both**	8.0695	4.9329	20
		bur	8.7281	4.9996	20
		etch	7.8555	5.4817	20
		no	7.1191	5.7342	20
		Total	7.9431	5.2281	80
	Z	**both**	14.1026	4.3199	20
		bur	12.9678	4.0558	20
		etch	14.1966	4.4891	20
		no	16.7462	4.5130	20
		Total	14.5033	4.4860	80
	Total	**both**	11.0860	5.5027	40
		bur	10.8480	4.9800	40
		etch	11.0261	5.8963	40
		no	11.9327	7.0503	40
		Total	11.2232	5.8657	160

Tests of Between-Subjects Effects
Dependent Variable: MPA

Source	Type III Sum of Squares	df	Mean Square	F	Sig.
Corrected Model	2646.339(a)	15	176.423	8.995	.000
Intercept	20153.583	1	20153.583	1027.556	.000
AGENT	600.933	1	600.933	30.639	.000

COMP	1721.480	1	1721.480	87.772	.000
SURFACE	28.072	3	9.357	.477	.699
AGENT * COMP	28.380	1	28.380	1.447	.231
AGENT * SURFACE	58.928	3	19.643	1.002	.394
COMP * SURFACE	151.167	3	50.389	2.569	.057
AGENT * COMP * SURFACE	57.378	3	19.126	.975	.406
Error	2824.290	144	19.613		
Total	25624.212	160			
Corrected Total	5470.629	159			
a R Squared = .484 (Adjusted R Squared = .430)					

Post Hoc Tests

SURFACE

Multiple Comparisons
Dependent Variable: MPA
Tukey HSD

					95% Confidence Interval	
(I) SURFACE	**(J) SURFACE**	**Mean Difference (I-J)**	**Std. Error**	**Sig.**	**Lower Bound**	**Upper Bound**
both	**bur**	.2381	.9903	.995	-2.3060	2.7821
	etch	5.999E-02	.9903	1.000	-2.4841	2.6041
	no	-.8466	.9903	.828	-3.3907	1.6974
bur	**both**	-.2381	.9903	.995	-2.7821	2.3060
	etch	-.1781	.9903	.998	-2.7221	2.3660
	no	-1.0847	.9903	.692	-3.6287	1.4594
etch	**both**	-5.9987E-02	.9903	1.000	-2.6041	2.4841
	bur	.1781	.9903	.998	-2.3660	2.7221

	no	-.9066	.9903	.797	-3.4507	1.6375
no	**both**	.8466	.9903	.828	-1.6974	3.3907
	bur	1.0847	.9903	.692	-1.4594	3.6287
	etch	.9066	.9903	.797	-1.6375	3.4507
Based on observed means.						

Homogeneous Subsets

MPA Tukey HSD		
	N	**Subset**
SURFACE		**1**
bur	40	10.8480
etch	40	11.0261
both	40	11.0860
no	40	11.9327
Sig.		.692
Means for groups in homogeneous subsets are displayed. Based on Type III Sum of Squares The error term is Mean Square(Error) = 19.613.		
a Uses Harmonic Mean Sample Size = 40.000.		
b Alpha = .05.		

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